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DESIGNERS AND
MANUFACTURERS OF
ADVANCED PRODUCTION
SYSTEMS FOR COMPOSITES

FINAL REPORT
DEVELOPMENT OF CONTINUOUS FORMING AND
CURING TECHNIQUES FOR PRODUCTION OF
CIRCULAR STRUCTURAL COMPOSITE SHAPES
FOR SPACE VEHICLE APPLICATION

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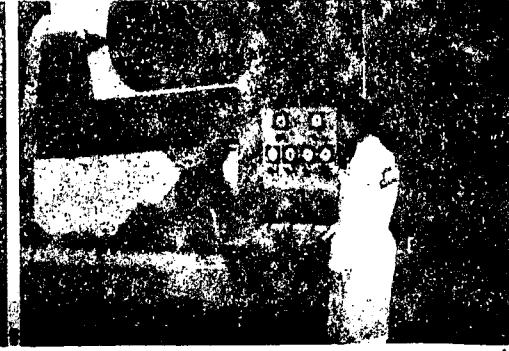
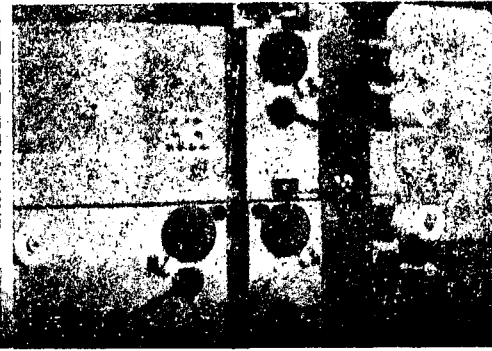
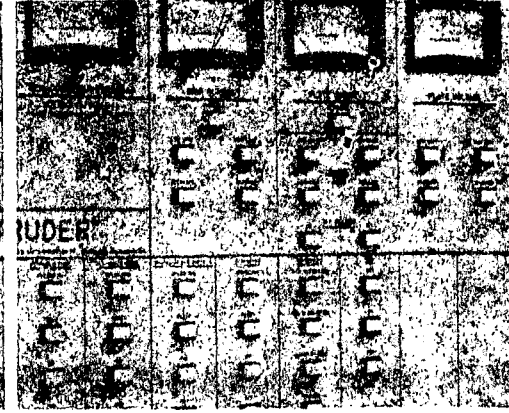
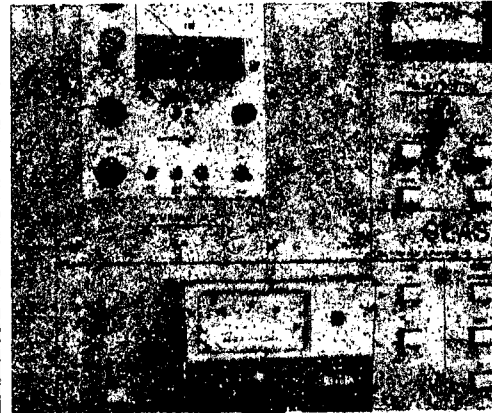
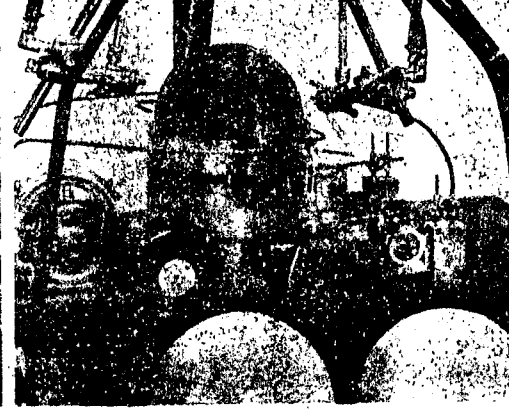
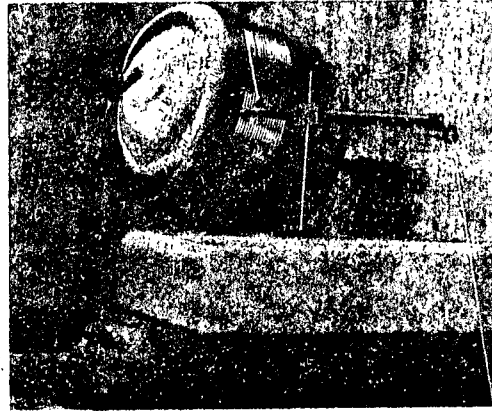
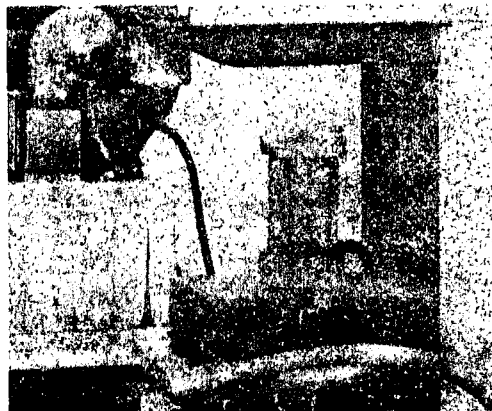
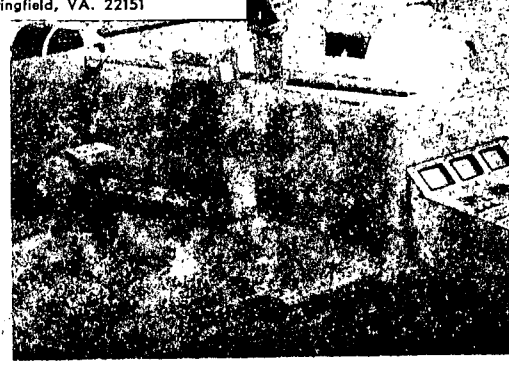
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PHONE (213) 534-4900



FINAL REPORT

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CURING TECHNIQUES FOR PRODUCTION OF
CIRCULAR STRUCTURAL COMPOSITE SHAPES
FOR SPACE VEHICLE APPLICATION

Contract No. NAS8-26900

June 24, 1971 - December 14, 1972

Sponsor:

George C. Marshall Space Flight
Center, NASA, Marshall Space
Flight Center, Alabama 35812

Prepared by:

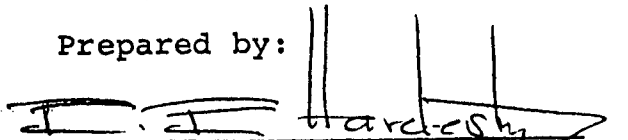

E. E. Hardesty
Program Manager
Goldsworthy Engineering, Inc.

TABLE OF CONTENTS

	<u>Page</u>
Abstract	iii
1.0 Introduction	1
2.0 Technical Discussion	3
2.1 Task Definitions and Philosophy	3
2.2 Phase I	5
2.3 Phase II	19
2.4 Phase III	28
2.5 Phase IV	33
3.0 Conclusions and Recommendations	39
4.0 References & Acknowledgements	44

TABLE OF ILLUSTRATIONS

Fig. 1A:	The Specified Hat Section	6
1B:	Layup Sequence	6
Fig. 2	Graphite Epoxy. Layup Orientations	18
Fig. 3	First Trial Run to Determine Possible Problem Areas	21
Fig. 4	Conclusion of Initial Run	22
Fig. 5	Circular Hat Sections	23
Fig. 6	Circular Channels and Angles	24
Fig. 7	Circular Zees and Widebase I	25
Fig. 8	Circular Bulb Angles and Tees	26
Fig. 9	One-piece Pultruded Structural Frame	27
Fig. 10	Four 12-foot Lengths of Graphite/Epoxy Hat Section	34
Diagram 1	Phase One Temperature Conditions	29
Diagram 2	Phase Two Temperature Conditions	30

ABSTRACT

The objectives of this development program were to devise practical and economical means for producing (straight and) circular standard sub-structural shapes and profiles from composite materials. The upcoming needs of the space vehicle program for cylindrical structures, fuel tanks, etc. has generated a requirement for circular structural members to be used as frames, skin stiffeners and the like. Structural composites are favored over metals for a number of such applications on two counts; weight and cost.

At the present time those ring-structure applications envision the use of standard profiles; hat sections, H and I shapes, etc. But to fabricate such profiles into complete rings, varying in diameter from twelve to more than twenty feet, from fiber-reinforced resin matrices, would - with conventional methods - require the use of full-sized static molding tools in which to lay up the necessary lamina layers and their individual orientations - by hand - to form that ring. Which would then be cured in an oven or autoclave to produce the finished full-diameter circular structure.

From this contractor's extensive, and - with regard to the use of dielectric heating/curing - unique experience and production history in the technique of pultrusion it was proposed that full-size structural rings might be made using this method.

This final report summarizes the results of work in successfully developing this new method, under the referenced NASA contract.

1.0 INTRODUCTION

1.1 Development work under this contract was divided initially into three phases, which are summarized:

a) Phase I:

Utilizing basic pultrusion equipment, - research, development, tooling design and fabrication procedures will be performed to produce a straight continuous hat section 1.5 inches wide, 1.5 inches high, with flanges 0.75 inches wide. Cap and flanges shall be 3/16 inches thick, with web sections 3/32 inches thick. Configuration development shall be carried out using fiberglass roving and fabric tapes; the deliverable item will be a graphite/epoxy hat stiffener of this cross section, 12 feet in length, with a fiber volume of approximately 60%.

b) Phase II:

Research, development, and tooling design will be performed to modify pultrusion equipment to generate the capability for continuous impregnation, forming, curing, and extraction of curved, fiber-reinforced hat sections, of the cross-section configuration described in Phase I. Fiberglass roving and fabric tapes, impregnated with epoxy resin, shall be utilized in the development and production of a 12 foot long section, formed to a 10 foot radius.

c) Phase III:

Research will be performed to determine the feasibility of using high temperature resin systems in conjunction with continuous dielectric curing and forming techniques. Use of Polyquinoxylene, Polyimide, Polyphenylquinoxylene, or other appropriate systems shall be considered. A polymeric system capable of service at temperatures of at least 600°F shall be selected. The demonstration component shall be approximately 5 feet in length, and of the cross section described in Phase I. (Straight. Not circular).

- d) Phase IV: (Added, at the successful conclusion of the three preceding Phases)

Utilizing tooling developed under Phases I and II, fabricate the following pultrusion components, using Modmor Type II Graphite fiber, 7 tows per inch, impregnated with Whittaker Corporation 5206 hot-melt epoxy:

1. 4 - 12' lengths of graphite-epoxy straight hat-section stiffener (48' min. total)
2. 1 - 12' arc length of curved (10' R) hat-section stiffener.

1.2 All items, of all Phases, were successfully concluded with the single exception of the very last item: #2 of Phase IV. This report describes that work, in summary form, but with emphasis on several highlights - in the form of unanticipated problems as well as achievements - which may be of technical interest.

1.3 It should probably be explained that this R & D effort was originally intended for funding under a CPFF-type contract. Which was later changed, with our concurrence, to a FFP contract. As it turned out, most of the contract remained reasonably within that fixed budget except for Item 2 of Phase IV. Which overran 289% (Ref. 4-16).

2.0 TECHNICAL DISCUSSION

2.1 Task Definitions and Philosophy

- 2.1.1 Pultrusion, which has been employed for over twenty years in the commercial production of structural bars, shapes and profiles from glass-reinforced/polyester composites, is becoming increasingly attractive as a potential manufacturing method for producing aerospace substructural shapes. Particularly from epoxy-impregnated, high-specific-efficiency advanced composites. The pultrusion technique is fairly straightforward; it is a continuous production method which pulls in fibrous and/or woven filamentary reinforcing media, arranges them into their respective orientation and inter-lamella relationships, impregnates them with a liquid thermosetting resin (or bypasses that in-line station if preimpregnated materials are being drawn in), fully preheats the laminate with dielectric energy, then draws it into a heated - forming, compacting, surface-smoothing - externally-heated metal die. From which it emerges usually fully cured. Except for some epoxies and all polyimides, which emerge cured to an advanced, hard B-stage, but usually requiring a subsequent post cure to achieve complete polymerization of those binder resins.
- 2.1.2 This contractor produces a standard line of production pultrusion machines, named "Glastruders." Since there are always from one to four Glastruders in our shop at any given time in the process of being fabricated and assembled, at least one of these is usually available for our own use in conducting experiments looking toward advancing the state of the art in this technique. Thus, although we do not accept orders for standard pultruded end products, we are able to utilize this equipment to undertake new and promising developments.
- 2.1.3 Consistent with this policy, we have elected - over the last several years - to use our existing pultrusion knowledge to take on original, first-time tasks. But limited to those which appeared to have considerable future potential and promise, as will be understood. This philosophy has led to a number of innovative undertakings, most of which have resulted in developing new means for producing structural shapes by this method. For example, the first of such undertakings, using advanced composites, sought to produce 3/16 in. boron/epoxy solid round rod,

essentially continuously. This took place some five years ago and was done for Boeing.* The developed method resulted in the continuous drawing in of boron filaments (actually 1/8 in. wide, preimpregnated - Narmco 5505 epoxy - tapes) forming, compacting and curing them into a continuous solid rod. Based on our earlier experiences with curing epoxy resins dielectrically (glass/epoxy filament wound pressure pipe) we used microwave energy; 2-1/2 KW at 2450 MHz. Which was definitely a First in this type of application. Another example, later, saw the continuous production of .125 in. thick by 2.00 in. wide flatbar from graphite/epoxy prepreg. Following those initial developments, various shapes have since been pultruded, and from several different materials. Among the more notable have been several "hybrid", composite-composites, containing glass/graphite, glass/boron, PRD-49/boron, glass/PRD-49, etc.

- 2.1.4 That earlier experience, in pultruding simple rods and flatbars - from boron/epoxy and graphite/epoxy, respectively - generated a reasonable level of confidence that the hat section shape called for in this contract could be successfully pultruded, although it hadn't actually been done before.
- 2.1.5 Far more difficult, it was thought, at the outset, would be the task of pultruding the specified hat section (or any similar profile) into a ring; in this case one which would be 20 feet in diameter. Pultrusion, per se, is somewhat analogous to the extrusion of metals or thermoplastics. Which, almost by definition, describes a straight-out, straight line exudation of solidifying materials. None of which have, to our knowledge, ever been permanently formed into a ring right as they were emerging from their initial shaping bushing or die. However, because these fiber-reinforced shapes are always pulled from a forming/curing die - with the fibers usually having sufficient tensile strength to withstand most pulling and friction forces, even irrespective of whether they are imbedded in an uncured resin matrix or not - such a process seemed at least feasible. The critical area being that discrete point of transition between the in-pulling, of straight-line, loose and disassociated filamentary reinforcements and their immediate transposition into a radius of curvature.

* Who has used that initial experience to qualify for a current Air Force contract seeking to produce structural shapes from advanced composites, using microwave for final cure.

- 2.1.6 It was decided, rather obviously, that this tangential translation would have to take place while the in-feeding materials were still soft and compliant. This meant that the primary compaction/curing die would then also serve as a reforming force to shift the laminate's stacked lamellas - in plane - to compensate for their immediately altered, section geodesic path length; inner to outer radii of the assumed curvature.
- 2.1.7 Based only upon this background of light experience and method concepts we went to work on satisfying the objectives of this contract.
- 2.2 Phase One
- (Produce, continuously, a straight hat-section. Initially from fiberglass, but with the deliverable end item from graphite/epoxy. 1 piece, 12 feet long)
- 2.2.1 Because the commercial/industrial production of structural shapes by pultrusion nearly always uses various forms of fiberglass, together with either a polyester - mostly - or epoxy resin, it was only necessary that we run enough of these conventional materials to accomplish initial "tool proofing".
- 2.2.2 On July 6th, 1971, Mr. H. M. Walker, NASA, (S & E - PT-MXS) visited this facility for the purpose of discussing the details of this contract during its initiating period. (Ref. 4.1). Concerning the specified hat section's general dimensions - 1.5 inches wide, 1.5 inches high, with flanges 0.75 inches wide, and with both cap and flanges 3/16 inch thick, but with web sections only 3/32 inches thick - it was decided that adequate corner radii should be also specified. This, to avoid the sometimes difficult to overcome inside-corner starving and outer-edge resin concentrations usually resulting from very sharp corners. In general, it was decided to use 1/8 in. nominal radii, except for the "toe" of each flange:

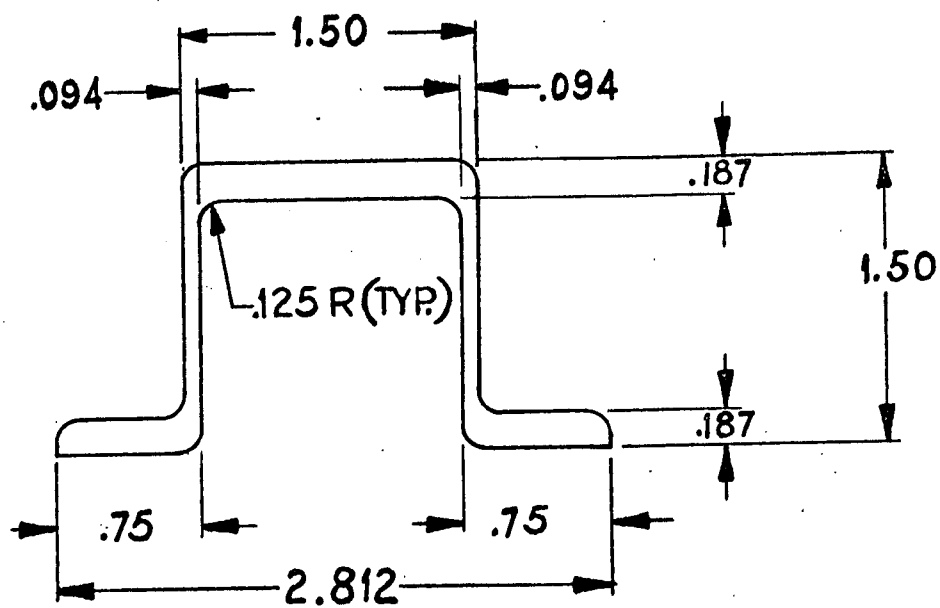


FIG. 1A - THE SPECIFIED HAT SECTION

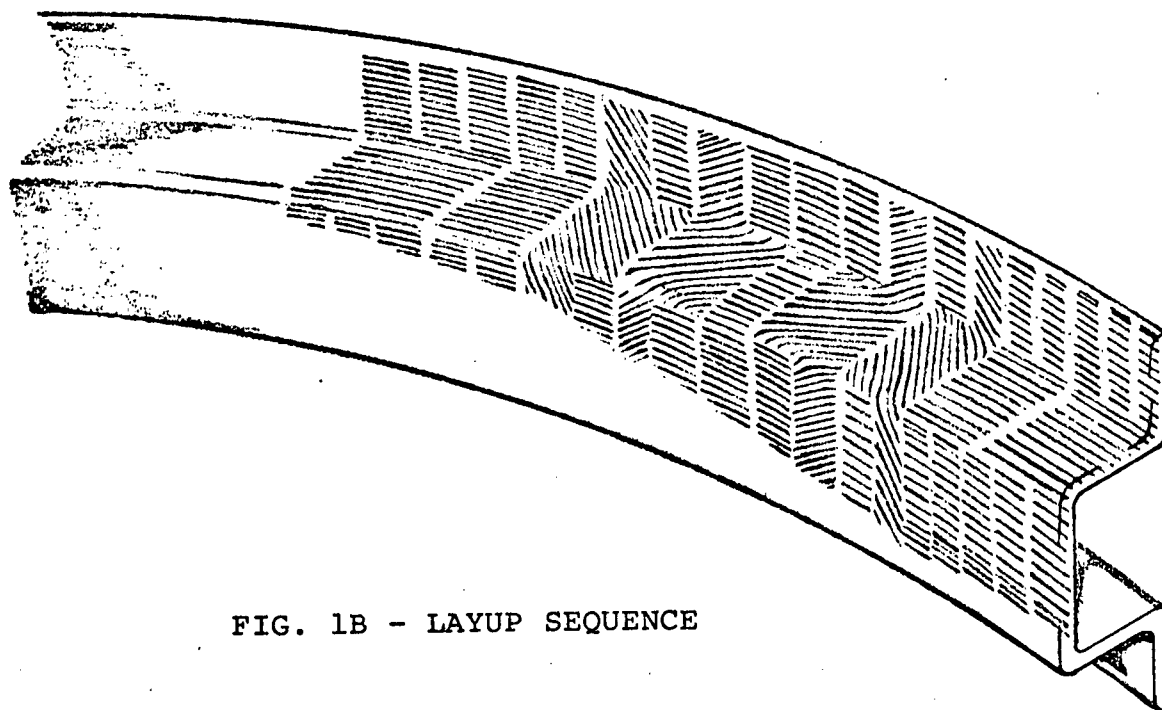


FIG. 1B - LAYUP SEQUENCE

- 2.2.3 During contract negotiations it had been decided that, for Phase II, we would design and build only the actual special hardware needed for forming a 20-foot diameter ring. Other, ancillary equipment such as the basic hydraulic power unit, the radio-frequency generator and resonant chamber, machine controls, etc. would be borrowed from in-house "Glastruder" inventories for this purpose. Some of the larger components were machined in vendor's shops. (Ref. 4.2)
- 2.2.4 During the initial "tool proofing" of the Phase I/III (straight) hat-section die, it was discovered that the die had been ground to a taper along its longitudinal axis, causing recurring jamming of the through-pulling stock. This taper was missed in our receiving inspection who checked the individual die halves, but overlooked the assembled-halves taper. Such a taper, if more than only a few thousandths of an inch, will render a pultrusion die inoperable; if the taper is downstream, a laminate will be excessively wedged; if upstream, it will usually tear the entering material. The die had to be reworked, which meant regrinding, re-(hand) micro polishing and re-chroming. (Ref. 4.3)
- 2.2.5 Following those initial, tool-tryout runs with glass-fabric/epoxy prepreg material, it was decided to make several additional runs using a hybrid laminate consisting of a glass fabric outer surface - resulting in .032 in. all glass webs - but with the cap and flanges filled out (to a 0.032 in. core) with graphite/epoxy tape. This, to determine what would be required to feed in linear-tow graphite tapes in a manner which would avoid lateral drifting and bunching; being essentially a function of proper upstream tooling. (Ref. 4.4). The only problems experienced had to do with the epoxy resins used by vendors for pre-impregnating both the glass fabric and linear-tow graphite tapes:
- 1) The majority of epoxy prepregs, with only a few exceptions such as Whittaker's 5206, have 4% to >8% excess volatiles. These are, for those particular resin systems, needed to provide sufficient flexibility/drapability to allow them to be formed, as well as afford the necessary tack to keep them in position, once laid down. In pultrusion, formability, per se, need only be sufficient to prevent longitudinal cracking or splitting when bent over a radius. As for tack, it obviously isn't needed in pultrusion.

When such excess volatiles exist, they must be mostly removed prior to final molding/curing. This is a rather pointless and time-consuming process step in pultrusion, of course. And, although low-solvent epoxies can be employed for Phases I and II (the latter being a liquid-impregnation, in-process function anyway) we have found no "high temperature" epoxy or polyimide, so far, which can be employed in Phase III which does not contain a relatively high percentage of volatiles, either in the form of residual solvents or as a byproduct of "pyrolytic polymerization," as in Pl3N polyimide. General Electric's "Germon L" polyimide is supplied, for example, with ~2% volatiles. G.E.'s local representative, K. A. Tennison is attempting to find out if G.E. can reduce volatiles to something in the order of .1-.3%, perhaps by pre-staging.

2) Excess resin. Prepregs, for hand (or machine) layup and autoclave/hydroclave molding have historically been supplied with 20-35% excess resin. This is, during conventional cure, pressed out of the laminate and collected in dry "bleeder" lamellas placed over the laminate. It will be obvious that, although some slight amount of excess resin is desirable in pultrusion - as an effective hedge against resin starving, for example - anything over, say, 1-3% creates problems. Foremost among these problems is pure hydraulic shear, where the high viscosity of just-softened epoxy creates a drag force of considerable magnitude, sufficient to pull apart, in tension, the die-traversing uncured materials. And, since this excess can only be removed by squeezing the collected impregnates as they are drawn into the tapered die entrance, they exert (in the fashion of the perversity of inanimates) unpredictable muscles, acting to push the in-feeding materials out of line, by building up a viscous roll of drooling resin which shifts, haphazardly, around the profiles's periphery. Efforts are being made to procure preimpregnates with near net resin content, but with only limited success so far; it is a new demand, apparently, and is being received by impregnators with some hesitation; even trepidation.

- 2.2.6 Because virtually all such substructural shapes and profiles - from advanced composites - will include bidirectionally-oriented tape layers, in addition to straight 0° longitudinals, and because the pultrusion process is essentially continuous, it was decided that such interspersed, off-axis, layers would have to be laid up by hand. Simply in order to build a sufficient length of assembled, oriented tape to make an economically practicable, continuous, run. And, because standard linear-tow preimpregnated tapes are rather fragile - being only superficially stuck together, contiguously, by their binder resin - it was decided to order that quantity of 3-inch tape representing the required yardage of specified $\pm 45^\circ$ layers (four) to be put up on a glass fabric, scrim cloth backing web. For better handling during layup.
- 2.2.6.1 This technique didn't work at all well. First pultrusions from graphite/epoxy split and - literally - fell apart at each scrim-cloth strata. Subsequent discussions with the vendor (Whittaker) didn't bring out any particularly strong reasons or hypotheses for the complete lack of adhesion between their (#5206 epoxy) matrix and the glass scrim. (Ref. 4.4).
- 2.2.7 The vendor had agreed to also lay up the required layers of 0° and $\pm 45^\circ$ tapes to form one assembled 3-inch wide, multi-layer, already assembled tape strip for feeding directly into a standard "Glastruder" pultrusion machine. As received, however, the two $\pm 45^\circ$ double layers were pretty sloppily arranged, with edge overlaps and gaps between adjacent 45° tapes of up to .068 in. wide. Since it was physically impracticable to attempt to peel all the layers apart in order to re-lay the $\pm 45^\circ$ diagonal pieces, that order of graphite/epoxy was used only for initial experimental, material-characteristics runs. A re-order was placed for a like quantity of replacement material, but without either scrim cloth or pre-laidup biased layers; we would thereafter do all our own layups. (Ref. 4.6).

Note: It will be fairly apparent that, if pultrusion is to achieve its inherent manufacturing cost savings it will need an accessory, up-stream mechanical device which will lay down - continuously - strips of preimpregnated tape at their required off-axis angles. Under that arrangement, rolls of 3-inch (or wider, for whatever developed widths are required) are simply drawn in, continuously, in their respective stacking order, with interleaving

of patches of skewed-angle tape laid down contiguously onto their respective adherend layers in proper sequence. This contract did not provide for the addition of such a continuous-laydown function, but its need soon became rather apparent from the fact it usually required some eighteen hours of hand layup to provide a >12 foot long multi-layer assembled tape, but only a few minutes to run it through the pultrusion process.

2.2.8 Pultruded sections often tend to emerge from the curing die with a matte, or frosty, surface finish. This is particularly true when using epoxy resins, which - during passage through the final (metal) curing die - are inclined to try to adhere to the die surfaces, in route, which results in the deposit of a light haze of cured epoxy on all die faces. Which thereafter acts to lightly abraid the surface of through-moving stock. This is not usually detrimental to the finished product, and, in fact, this lightly roughened surface can be beneficial in subsequent adhesive bonding. However, linear-tow graphite has, between each tow, linear ridges of resin. These cause a striping on die surfaces which, in turn, produces shallow longitudinal scratches on the finished stock. We attempted to remedy that surface condition - and eliminate a sometimes freezing of the through-moving stock - by employing a 5.5 in. wide Teflon-fabric slip tape, ("TX-1040" or "Armalon 406C") applied to both surfaces as the material entered the die.

2.2.8.1 This attempted improvement of the product's surface finish didn't turn out very well. As the assembled - dielectrically-preheated - laminate enters the final curing die it is subjected to instant and rather abrupt debulking. This wedging/squeezing action caused the Teflon fabric to buckle and wrinkle, with most wrinkles being thereafter practically ingested into the surface of the moving stock. Even the addition of considerable back tension, smoothing bars, etc. to the infeeding fabric had no appreciable effect at reducing wrinkles. Even further, since the top and bottom Teflon-fabric tapes effectively sandwiched the entering (uncured) laminate, all that extra epoxy - discussed earlier - could only escape out of the layup's two open edges; the hat section's "toe" area.

Therefore, since the epoxy prepregs we were using had to dispose of some 12% excess resin - in order to achieve the ~60% fiber volume specified - resin flowed in relatively high volume from both edges as they entered the die. These epoxies, being "hot melt", retain a comparatively high viscosity, even at the ~340°F pre-heat temperature. This caused a severe viscous-shear "washing" and displacement of individual graphite tows, simply from being pushed out by the force of the escaping resin.

2.2.9 When it was decided that we should assume the task of laying in the two pairs of $\pm 45^\circ$ layers, the first 12 ft. + length of graphite/epoxy hat section produced from our layup was pultruded with no operational problems. Except it assumed a quite uniform, longitudinal twist. This twist came to nearly 90° of rotation in the 12-foot length. We decided, in any event, to subject this full-length hat section to the normal 8-hour postcure anyway, just to determine if its twist would increase or could be decreased. The latter being based on the fact that, during postcure, all hat sections were restrained by a full length aluminum-plate "T" shaped, inside-shape-fitting tool. Following post cure, that hat section - upon removal from its postcure fixture - tightened its twist to ~180°; essentially double what it had been directly following its pultrusion.

2.2.9.1 Although it was pretty generally agreed, subsequently, that our layup sequence had probably been wrong, we decided to see if we could analyze for the reason(s) before we attempted any more (expensive) runs. That analysis, by our Dr. Brian H. Jones, (Ref. 4.8) provided the following conclusions:

This note will be directed towards a consideration of the requirements for attaining balance in laminated composite materials. The types of lamina coupling to be considered will be described in terms of:

- a) in-plane shear deflection
- b) out-of-plane bending and warping

The loading situations that give rise to the foregoing distortions may be considered as internally induced (differential thermal strains) externally induced (in-plane loading) or combinations of these.

The nature of the problem can best be described by reference to the well-known constitutive equation:

$$\begin{bmatrix} N \\ M \end{bmatrix} = \begin{bmatrix} A & B \\ B & D \end{bmatrix} \begin{bmatrix} e^o \\ k \end{bmatrix}$$

where the stress and moment resultants are given by N and M respectively and

$$(A_{ij}, B_{ij}, D_{ij}) = \int_{-h/2}^{h/2} Q_{ij} (1, z, z^2) dz \quad (1)$$

where Q_{ij} is the transformed stiffness matrix.

Design and Manufacturing Restraints On the A_{ij} Matrix

Because analysis techniques can only be realistically employed when the laminate behaves as an orthotropic material then it is evident that for such a condition:

$$A_{16} = A_{26} = 0$$

Since the A_{ij} are equal to the sum of the lamina Q_{ij} times the lamina thickness, then the only way an A_{ij} term can be zero is for either all the Q_{ij} to be zero or to have some positive and some negative. As Q_{ij} is derived from the orthotropic stiffnesses they are always positive and greater than zero. Hence A_{11} , A_{12} , A_{22} and A_{66} are always positive and greater than zero. On the other hand, Q_{16} and Q_{26} are zero for orientation of 0° and 90° and can be either positive or negative since these terms are defined in terms of odd powers and $\sin\theta$ and $\cos\theta$. In particular, Q_{16} , Q_{26} for a plus rotation is equal in absolute value but of the opposite sign from the Q_{16} , Q_{26} for a negative rotation.

Thus to ensure the laminate is specially orthotropic with respect to in-plane forces and strains, then for every lamina of a plus θ orientation there should be another lamina of the same orthotropic properties and thickness with a negative θ orientation.

Non-compliance with the foregoing requirement is not too significant from a manufacturing point of view, provided the stacking sequence is symmetric about the mid-plane (see Ref. 1)

Design and Manufacturing Restraints on the B_{ij} Matrix

If each $B_{ij} = 0$ then a major simplification in both analysis and manufacture occur since the bending and stretching problems are uncoupled.

The terms in the $[B]$ matrix are obtained as a sum of terms involving the $[Q]$ matrices and squares of the z coordinates of the top and bottom of each ply. Since the B_{ij} are thus even functions of the h_k , they are zero for laminates which are symmetrical with respect to z .

Thus each term B_{ij} is zero if for each lamina above the mid-plane there is an identical ply (in properties and orientation) located the same distance below the mid-plane.

This class of mid-plane symmetric laminates is an important class from a manufacturing point of view, since a non-symmetric situation causes undesirable warping due to in-plane loads induced by thermal contractions occurring during processing. With mid-plane symmetric laminates, thermal contractions introduce mid-plane strains (and in-plane loads) but they do not introduce bending.

Constitutive Equations for $B_{ij} = 0$

From equation (1) for $B_{ij} = 0$ is obtained

$$[N] = [A] [e^\circ]$$

$$[M] = [D] [k]$$

For the case when $A_{16} = A_{26} = 0$ then the equations become:

$$\begin{bmatrix} N_x \\ N_y \\ N_{xy} \end{bmatrix} = \begin{bmatrix} A_{11} & A_{12} & 0 \\ A_{12} & A_{22} & 0 \\ 0 & 0 & A_{66} \end{bmatrix} \begin{bmatrix} e_x^\circ \\ e_y^\circ \\ \gamma_{xy} \end{bmatrix}$$

Similarly for $D_{16} = D_{26} = 0$

$$\begin{bmatrix} M_x \\ M_y \\ M_{xy} \end{bmatrix} = \begin{bmatrix} D_{11} & D_{12} & 0 \\ D_{12} & D_{22} & 0 \\ 0 & 0 & D_{66} \end{bmatrix} \begin{bmatrix} k_x \\ k_y \\ k_{xy} \end{bmatrix}$$

Significance of the D_{ij} Terms

The D_{ij} terms are defined in terms of the Q_{ij} and the difference between the third power of the z coordinate at the layer top and the layer bottom; it can be shown that the D_{11} , D_{12} , D_{22} and D_{66} are always positive. The D_{16} and D_{26} terms are zero if all of the lamina are oriented at either 0° or 90° . Furthermore, the D_{16} and D_{26} terms are also zero if for every layer oriented at $(\pm\theta)$. This however, means that $B_{ij} \neq 0$. Actually for all except 0° (only) 90° (only) and $0^\circ/90^\circ$ laminates, D_{16} and D_{26} are not zero

for any mid-plane symmetric laminate. These terms do however become small for large number of (± 0) laminates and this justifies the general approach taken in analysis, which is to assume that

$$D_{16} = D_{26} = 0.$$

As a generality, the D matrix is not too influential on the processing problem.

Significance of Material Type and Number of Layers

Reference 2 considers the bending-extensional coupling in laminated plates under traverse loading and amongst other things determines the severity of the coupling effect.

Thus, the extent of the coupling effect depends on the number of plies in the composite and on the degree of anisotropy in the individual layers as determined by the ratio F , where $F = E_{11}/E_{22}$. The coupling coefficients are inversely proportional to the number of layers P .

Some results are given for glass/epoxy ($F=2.9$) and graphite/epoxy ($F=40$) and it is evident that for $P > 5$, the behavior is almost the same as for $P = \infty$.

Comparison of the Foregoing Conclusions with Practice

The conclusions derived during the course of this investigation were applied to the observed behavior of both glass/epoxy and graphite/epoxy hat stringer sections by pultrusion. The layup patterns are shown in Figure 2 for two glass/epoxy and two graphite/epoxy situations. Observations are as follows:

Glass-Epoxy

Case I. The layup pattern is not symmetrical about the mid-plane and so $B_{ij} = 0$. This should result in bending (since the layup is specially orthotropic). Inspection of the section indicated bowing consistent with the conclusions drawn. Since the effect of the

45° lamina is to increase the effective coefficient of thermal expansion ($\alpha_{\text{glass}} = 2.7 \times 10^{-6}/^{\circ}\text{F}$; $\alpha_{\text{epoxy}} = 40 \times 10^{-6}/^{\circ}\text{F}$) in these lamina, the bowing should be concave on the top of the cap, which is the case.

Case II. The layup is not symmetric about the mid-plane and so $B_{ij} \neq 0$. Also A_{16} and $A_{26} \neq 0$. Although no long sections were available for inspection evidence of "skewing" was observed, as should be the case if the conclusions drawn from this investigation are correct.

Graphite Epoxy

Case III. This pattern is both unsymmetric about the mid-plane and has +45° layers. Hence $B_{ij} \neq 0$ warping must occur under thermal straining. This was observed to be the case to a marked degree.

Case IV. This pattern does not produce $B_{ij} \neq 0$ since the layers are not equally oriented ($\theta_k = \theta_{-k}$). However, the requirement is not met by the distance of one layer only and should therefore produce less warping than in Case III. This was in fact found to be true.

Conclusions

1. To ensure no warping in composite structures subjected to thermal processing, the layup pattern should be such as to be identical about the mid-plane ($B_{ij} = 0$).
2. From the point of view of enabling analysis to be conveniently carried out, the layup should produce a specially orthotropic configuration.
3. The bending/shearing coupling cannot be completely eliminated for a general class of laminates, but this is not usually a problem for either design or manufacture.

4. To produce an example of balanced construction in Case IV layer 13 should be at $+45^\circ$ and layer 11 at -45° .

References

1. H. C. Schjelderup and B. H. Jones "Practical Influence of Fibrous Reinforced Composites in Aircraft Structural Design."
2. J. M. Whitney "Bending-Extensional Coupling in Laminated Plates Under Transverse Loading." J. Comp. Materials, 3, n1, 1969.

CASE I

1	_____	0°
2	_____	0°
3	-----	+45°
4	_____	
5	-----	-45°
6	_____	0°
7	_____	0°
8	_____	0°
9	_____	0°
10	_____	0°
11	_____	0°
12	_____	0°
13	_____	0°
14	_____	0°

CASE II

1	_____	0°
2	_____	0°
3	_____	0°
4	-----	+45°
5	_____	0°
6	_____	0°
7	-----	+45°
8	_____	0°
9	_____	0°
10	_____	0°
11	-----	-45°
12	_____	0°
13	_____	0°
14	_____	0°

CASE III

1	_____	0°
2	_____	0°
3	_____	0°
4	_____	0°
5	_____	0°
6	-----	+45°
7	_____	0°
8	_____	0°
9	_____	0°
10	_____	0°
11	_____	0°
12	-----	+45°
13	_____	0°
14	_____	0°
15	_____	0°

CASE IV

1	_____	0°
2	_____	0°
3	-----	+45°
4	_____	0°
5	-----	-45°
6	_____	0°
7	_____	0°
8	_____	0°
9	_____	0°
10	_____	0°
11	-----	+45°
12	_____	0°
13	-----	-45°
14	_____	0°
15	_____	0°

GRAPHITE EPOXY
LAYUP ORIENTATIONS

Fig. 2

As reconstructed by Dr. Jones, our longitudinal twist was, in fact, the result of a layup/layer sequence which saw the successive 45° layers laid on in a direct alternate sequence (+45°, -45°, +45°, -45°. As in "Case IV"). Whereas they should have been laid in a mirror relationship on either side of the laminate's centeroid mid-plane (+45°, -45° (CL) -45° +45°).

- 2.2.10 Succeeding layups - and runs - of graphite/epoxy (straight) hat sections thereafter quickly settled down the materials-feed/processing-parameter procedures until a suitable and acceptable 12-foot section was produced. This concluded the requirements for Phase I.

2.3 Phase Two

- 2.3.1 During that period when we were working on Phase I, orders had been placed for the necessary hardware and composite materials for use on Phase II; circular hat sections, 20 feet in diameter:

(Being: "tool design to modify pultrusion equipment to generate the capability for continuous impregnation, forming, curing and extraction of curved, fiber-reinforced hat sections. Fiberglass reinforcements, impregnated with an epoxy resin shall be used to produce one 12 foot long section, formed to a 10 foot radius")

- 2.3.2 As provided by the terms of the contract, certain of our standard machine and curing functions were drawn from existing "Glastruder" inventories and added to the circular pultrusion machine. These were; one standard radio-frequency generator (10 KW, 70 MHz) and its appended resonant chamber, one Vickers hydraulic pump/reservoir (20 h.p., 1500 psi) package power unit, one "Glastruder" main control panel, plus a number of control circuits, etc.

- 2.3.3 The philosophy employed in designing this circular-pultrusion machine ("tooling") resulted in fabricating two, 45° arcuate segments, the outer rims of which were machined to a 10-foot radius. Those machined surfaces also contained an indented central channel with undercut facings on either side, representing the specified-dimension hat section's outer-surface shape. Each of these arcuate sections were driven by a 15 h.p. hydraulic motor, pinioned to a sector ring gear, with each section rotating about a central, pivotal kingpost.

- 2.3.3.1 The principle, here, was to form a 20-foot diameter ring, but without resorting to a 20-foot complete wheel, since these partial circular segments would actually form a complete hat-section ring anyway, but are more amenable to the pultrusion of such rings because they will not

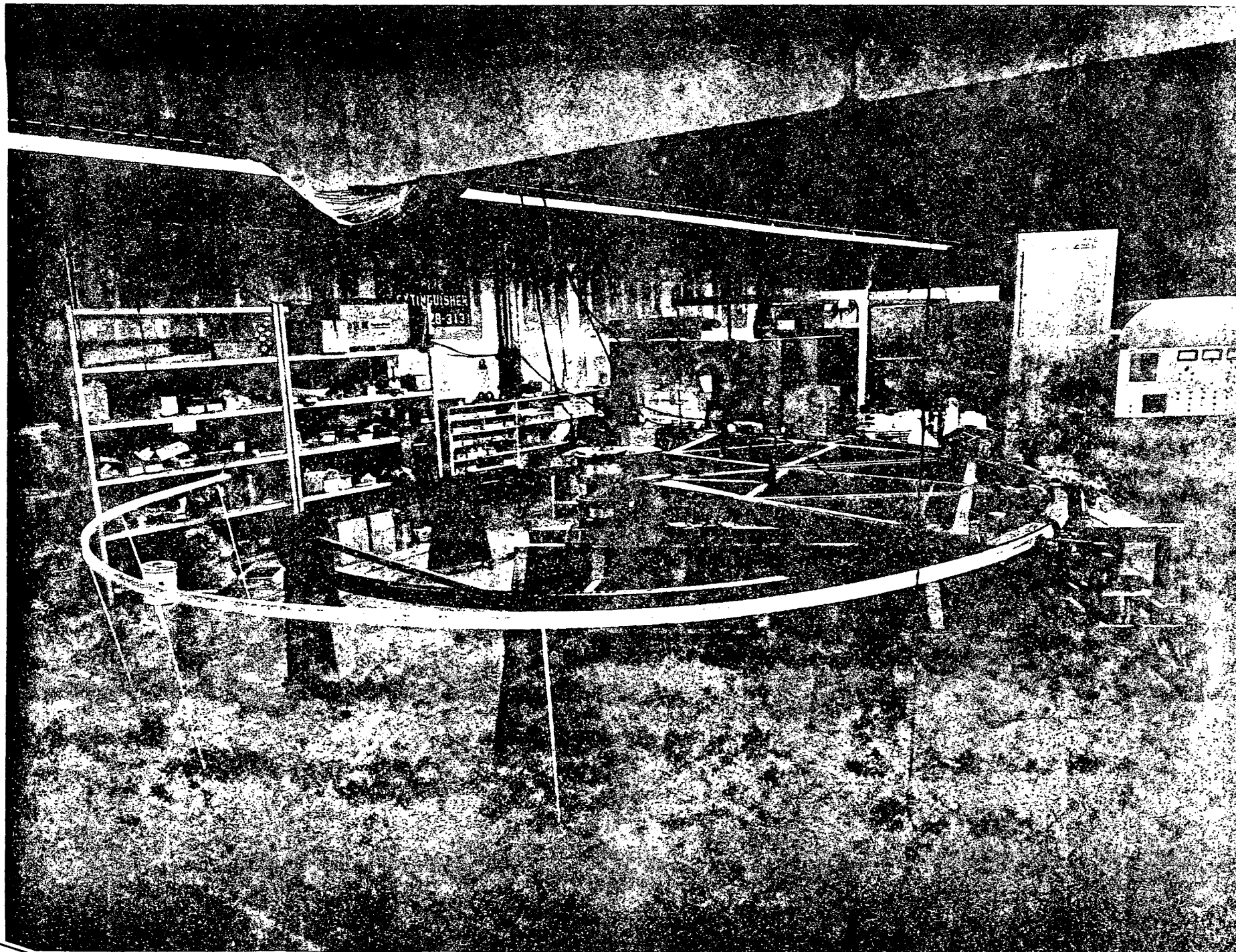
restrict the forming of a series of rings - in a continuous spiral - for sustained production. Which would not be possible with a full-diameter wheel, as will be understood. More to the point, perhaps, is that it is not really possible to mold a complete, 360° ring on a full-circle wheel because there must always be an added expendable leader - of some two feet in length - and a similar tail-off portion of about the same length. Which, since they cannot be overlapped, must be cut away, leaving a ~4 foot gap.

- 2.3.4 First trial runs were made using a special glass fabric - J. P. Stevens' #3743 full-strength-bias woven tape - purchased for this Phase II requirements but using a liquid polyester resin rather than an epoxy, simply to make startup and initial debugging somewhat easier. See Figs. 3 and 4 (Ref. 4.7). That very first run went off without a hitch, system-wise, with quite good stock being produced. But, when only ~190° of the circle had been completed it was discovered that the 5.5 in. woven-glass reinforcing tape had been rolled up from free ends; (unspliced) short lengths making up what had been reasonably assumed to be a continuous-tape roll of >200 feet. And, because we had made no provisions for such in line butt-splicing, the run had to be terminated.
- 2.3.5 Subsequent runs used (pre-spliced) glass-fabric tape and liquid epoxy resin (100 phr. Epon 826/90 phr NMA/0.1 phr DMP-30); preimpregnated Scotchply #1002 with Whittaker #5206 epoxy prepreg glass fabric; prepreg Scotchply core with wet-epoxy-impregnated #3743 glass surface tapes, etc. All of these various combinations worked quite well, although the Scotchply #1002 prepreg/wet-epoxy glass fabric generated a number of bubble-voids in both surfaces.
- 2.3.6 During the first of these experimental runs it was observed that temperatures varied over the length of each of the arcuate section's heated rims. Accordingly, we made a check of those rim's temperature profiles:

2.3.6.1 Rotating Die Temperature Conditions

DATA:

Energy Input:	6300 Watts
Die Weight:	490 Pounds
Temperature Measuring Device:	"Tempilstick:"Crayons



21

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FIGURE 3 FIRST TRIAL RUN TO DETERMINE POSSIBLE PROBLEM AREAS
GLASS FABRIC/POLYESTER



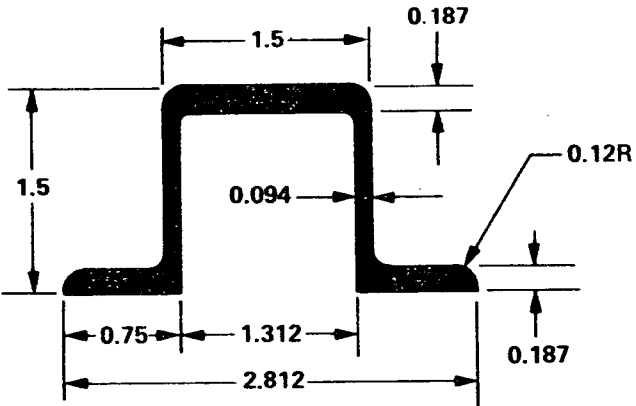
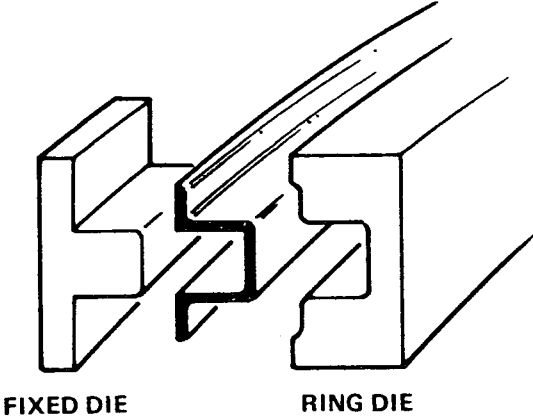
FIGURE 4 CONCLUSION OF INITIAL RUN. RF PRECURE
RESONANT CAVITY, EXTREME RIGHT

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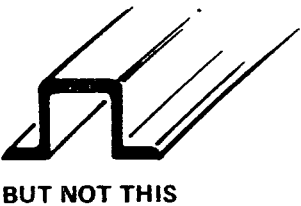
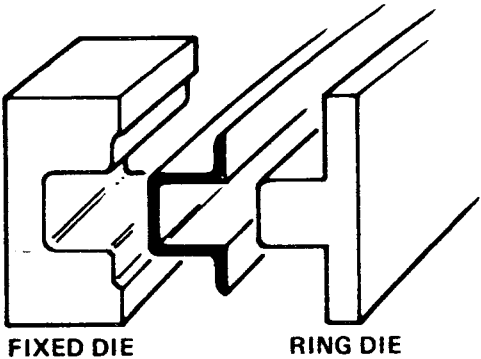


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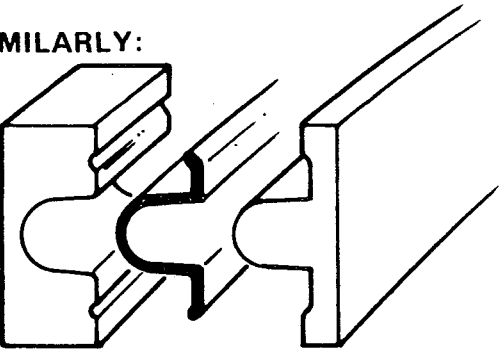
CIRCULAR HAT SECTIONS



ALSO:



SIMILARLY:



OR:

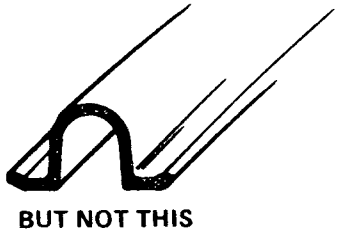
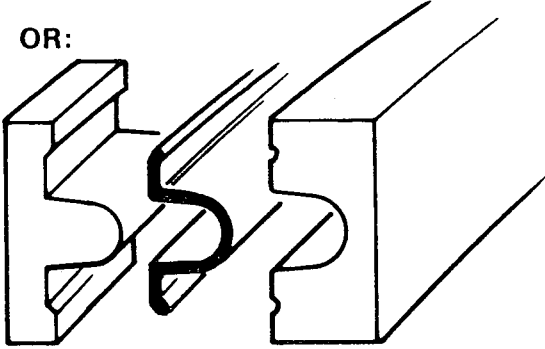


FIGURE 5

CIRCULAR CHANNELS AND ANGLES

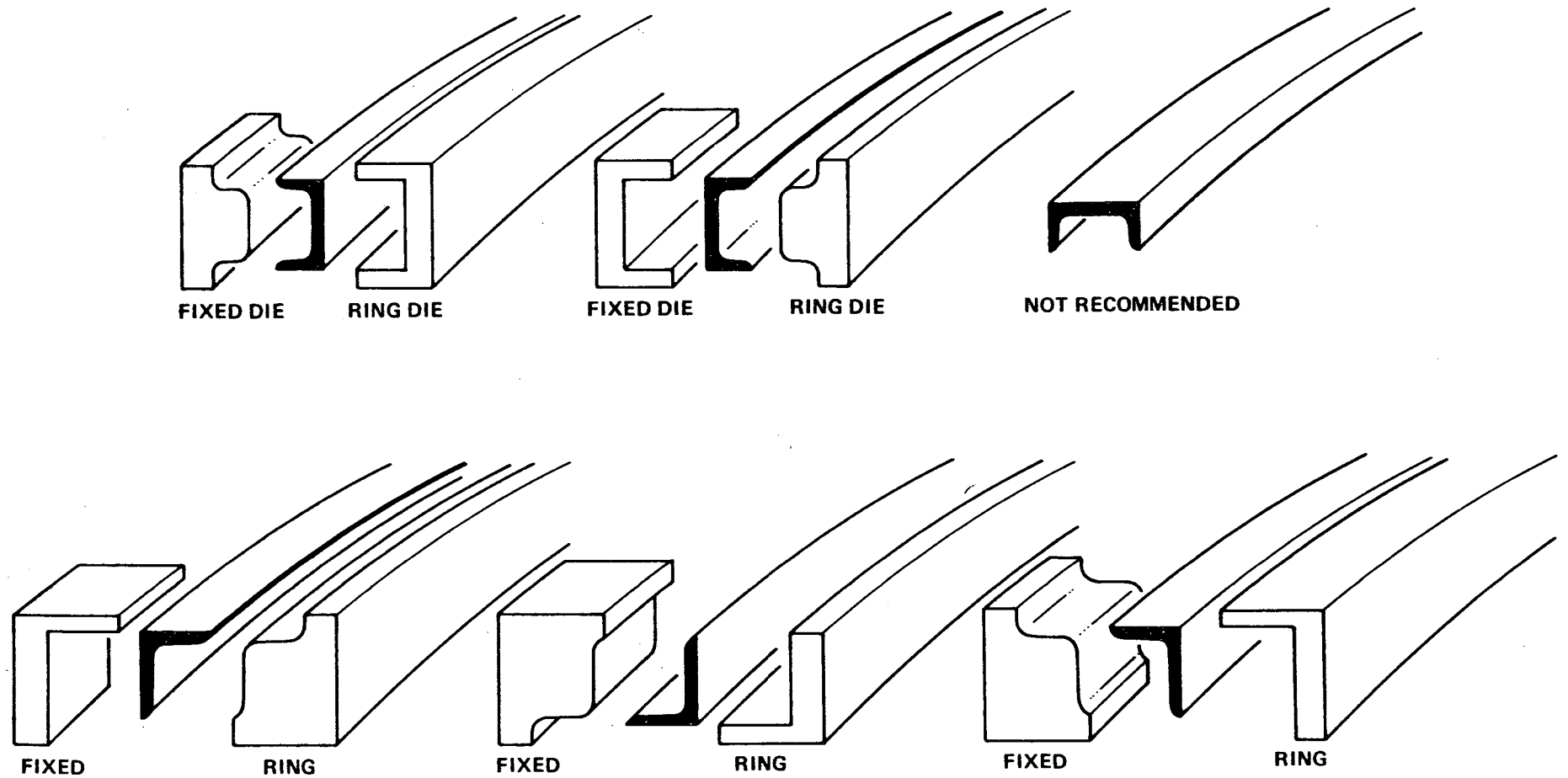


FIGURE 6

CIRCULAR ZEES AND WIDEBASE I

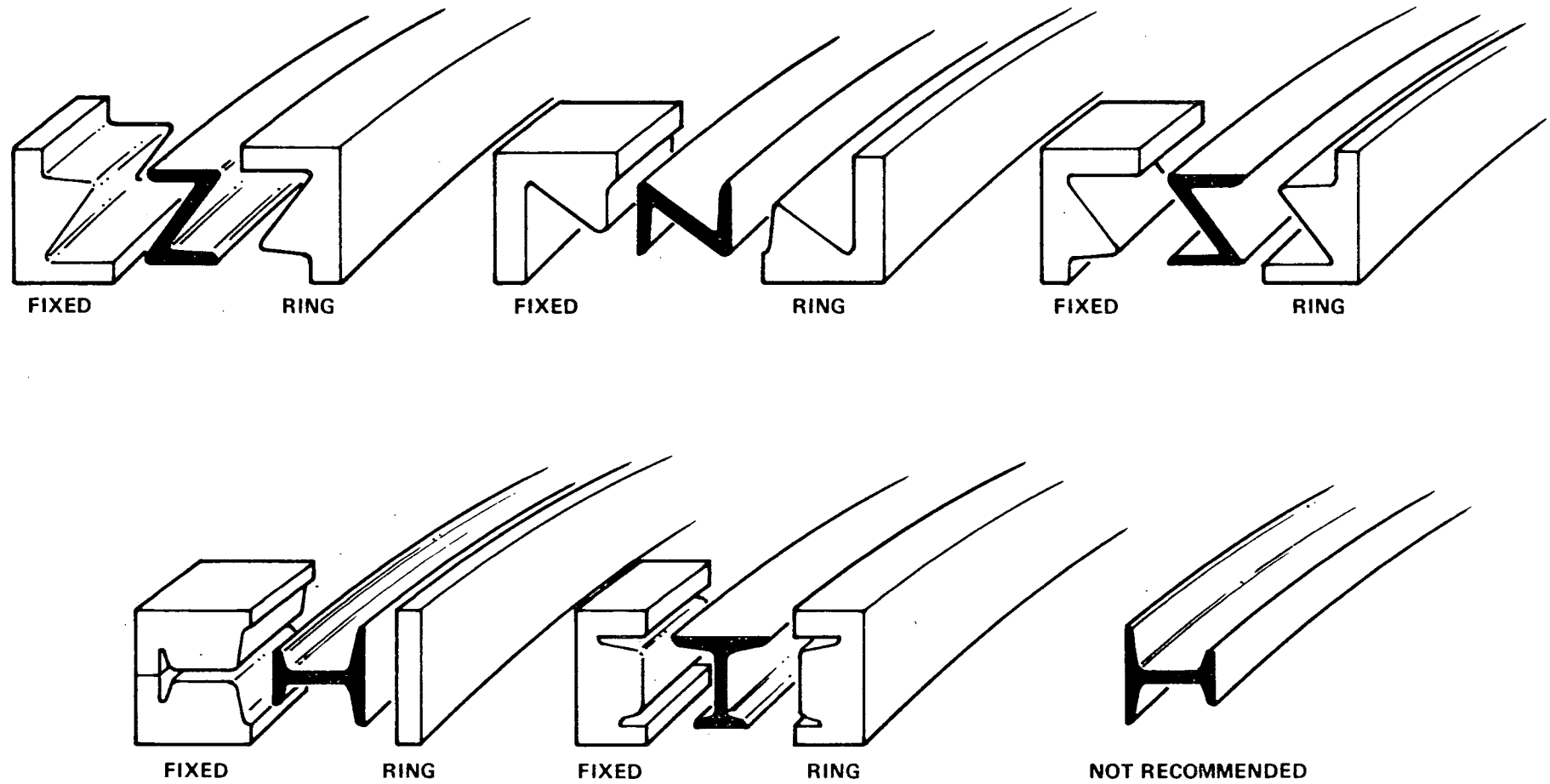


FIGURE 7

CIRCULAR BULB ANGLES AND TEES

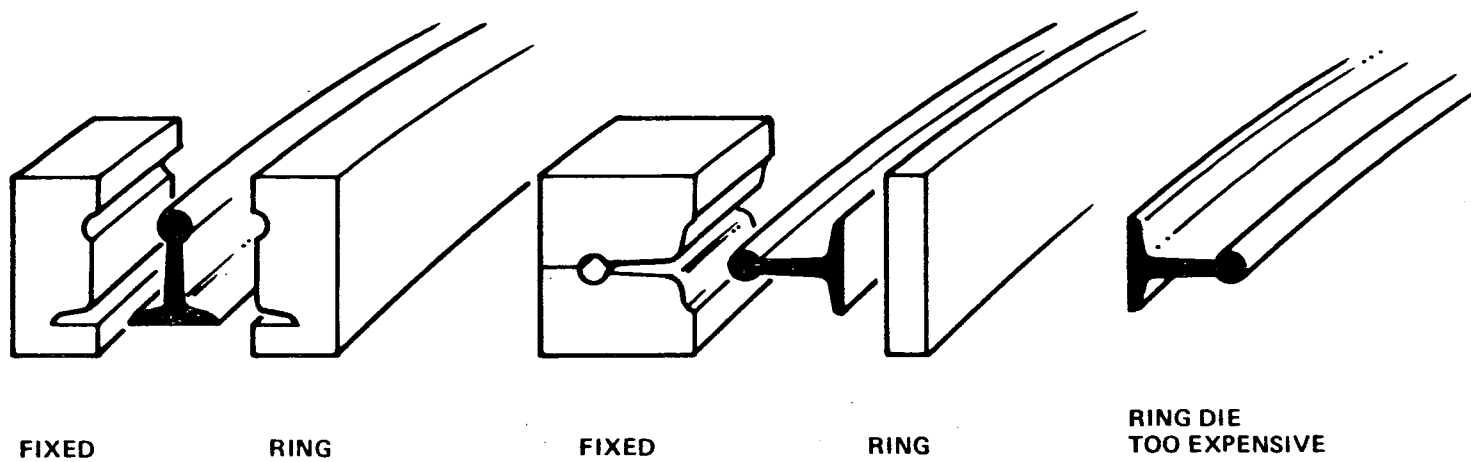
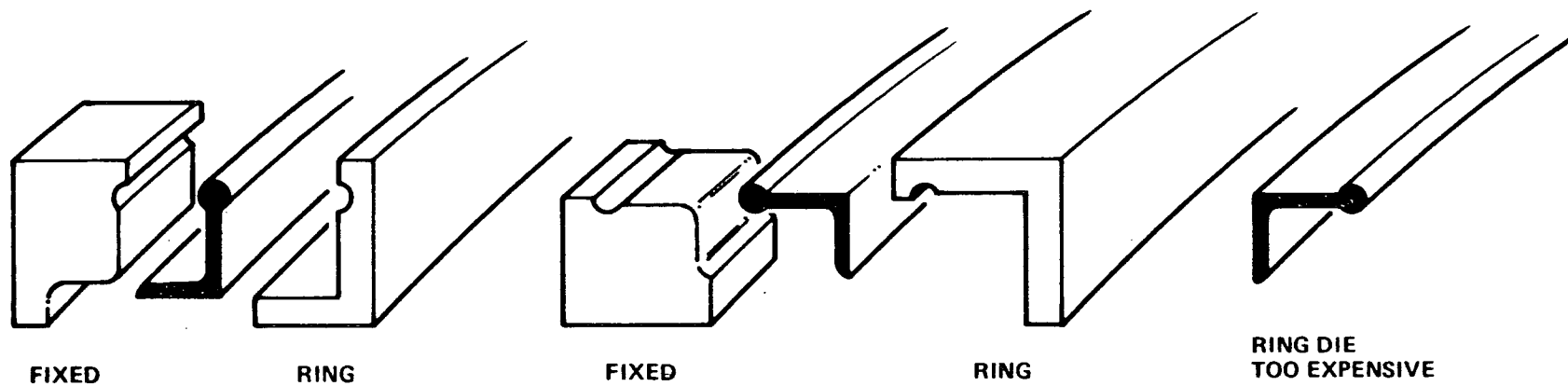
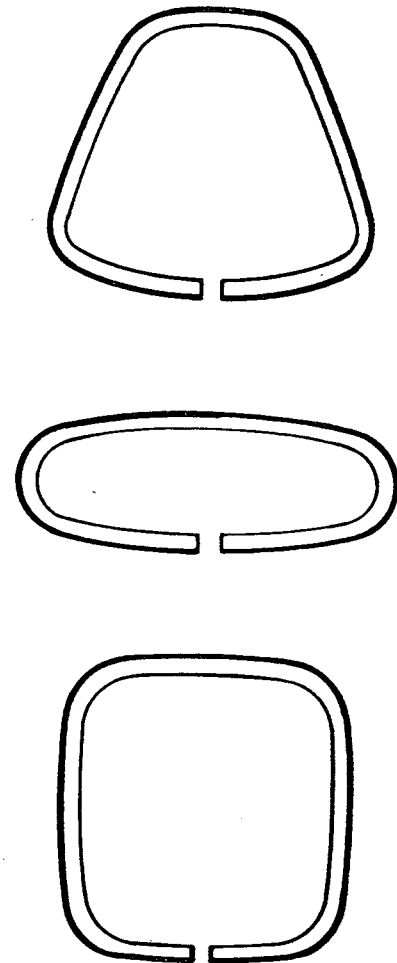
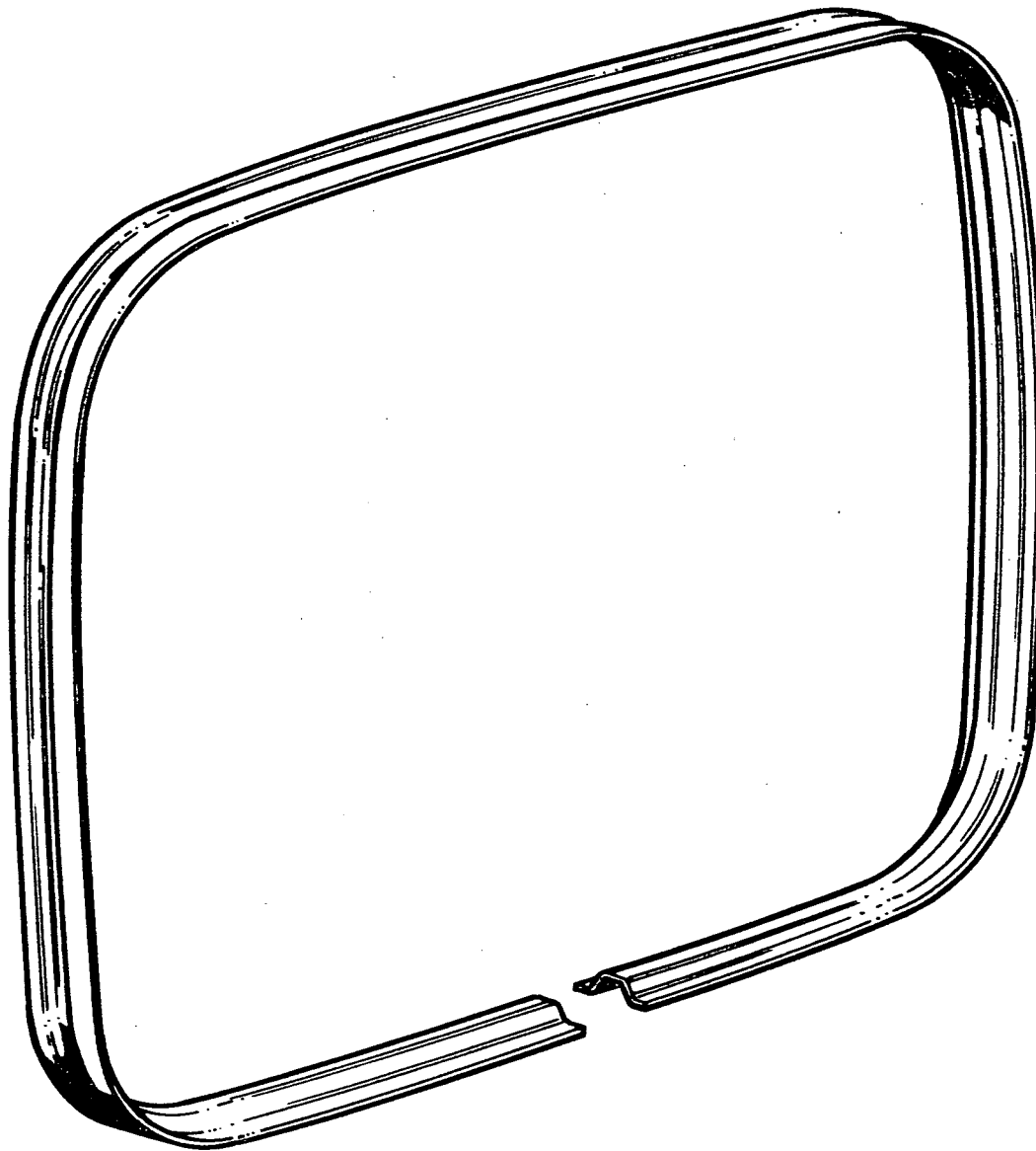


FIGURE 8



ROUNDED-SQUARE SHAPES

FIGURE 9 ONE-PIECE PULTRUDED STRUCTURAL FRAME.
EMPLOYING – FOR EXAMPLE – A HAT SECTION

Die temperature conditions were determined in two phases. Phase one was performed during the warmup period following a 20 minute power off heat stabilizing period. It was designed to determine the temperature profiles surrounding each heating element.

Phase two was performed after the working face of the die had exceeded 350°F at all points. It was designed to determine the overall die temperature profile.

The initial warmup period required one and one-half hours to bring the die to a temperature above 400°F except at the last inch of die at each end where the temperature exceeded 375°F. The power was then shut off for a period of 20 minutes during which time the temperature at the hottest points on the die was approximately 350°F. Power was turned on and the readings for phase one were taken approximately 5 minutes thereafter. Phase two conditions were reached 20 minutes after the resumption of die heating.

Phase one temperature conditions are illustrated in Diagram 1. Phase two temperature conditions are illustrated in Diagram 2.

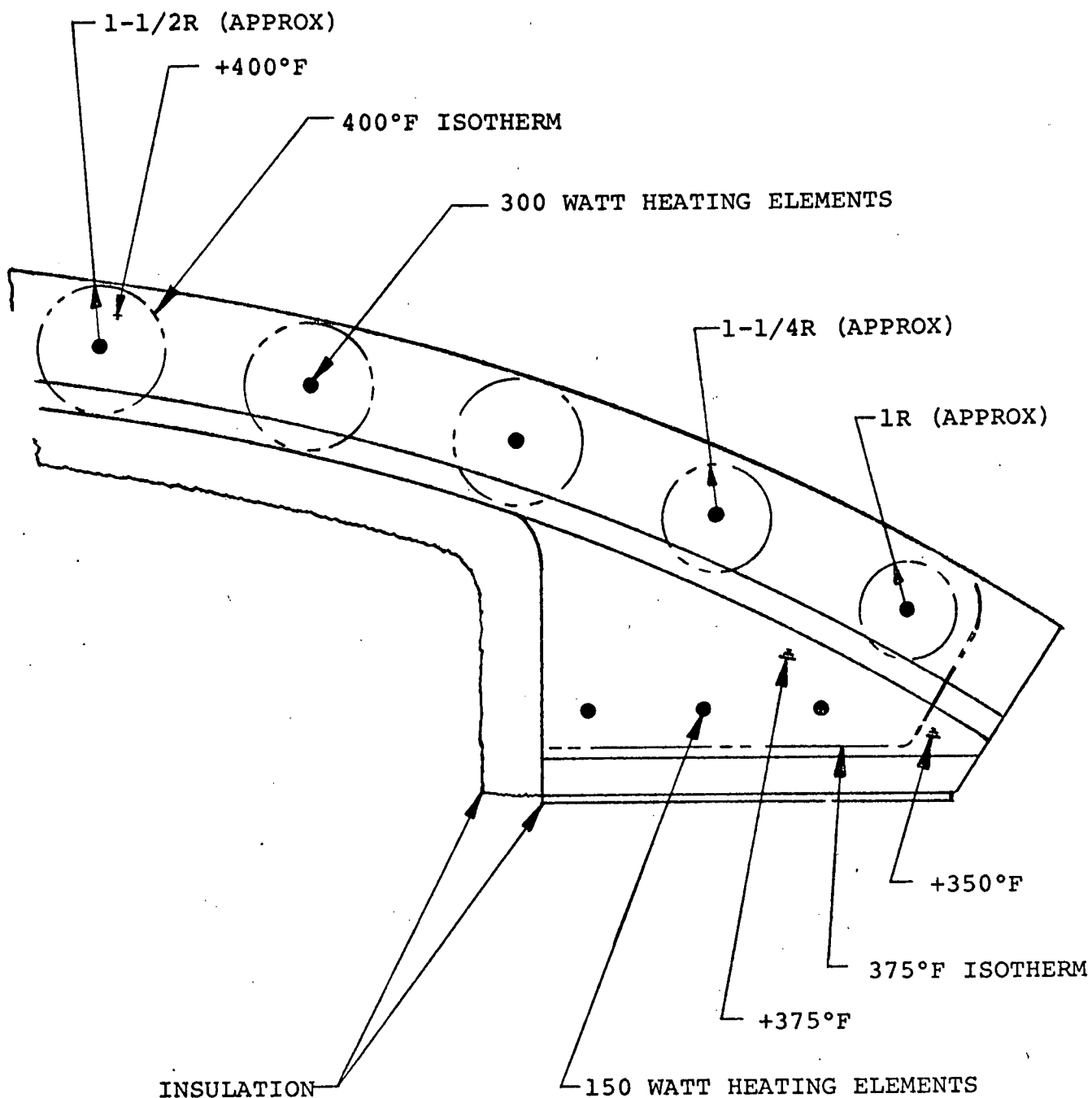
During the tests, the Sector temperatures were found to be approximately 200°F on the die mounting flanges but the tubular structure did not appear to be much above room temperature beyond nine inches from the flange.

Temperature variations and gradients, being originally somewhat larger than we thought acceptable, were caused by heat being drawn off at the connections between the ring's circular structural portion and its supporting radial spokes. Since the original heat-block pads could not be made thicker - without a complete disassembly of the equipment and machining off the faces of all connecting plates - the insulating pads were changed to Transite. Which, although still quite a ways from a reasonable optimum, nevertheless did tend to reduce the amplitude of the temperature variations.

- 2.3.7 The contract's specified deliverable item - one 12-foot length of glass fabric/epoxy hat section, made to a 10-foot radius - was pultruded and shipped.

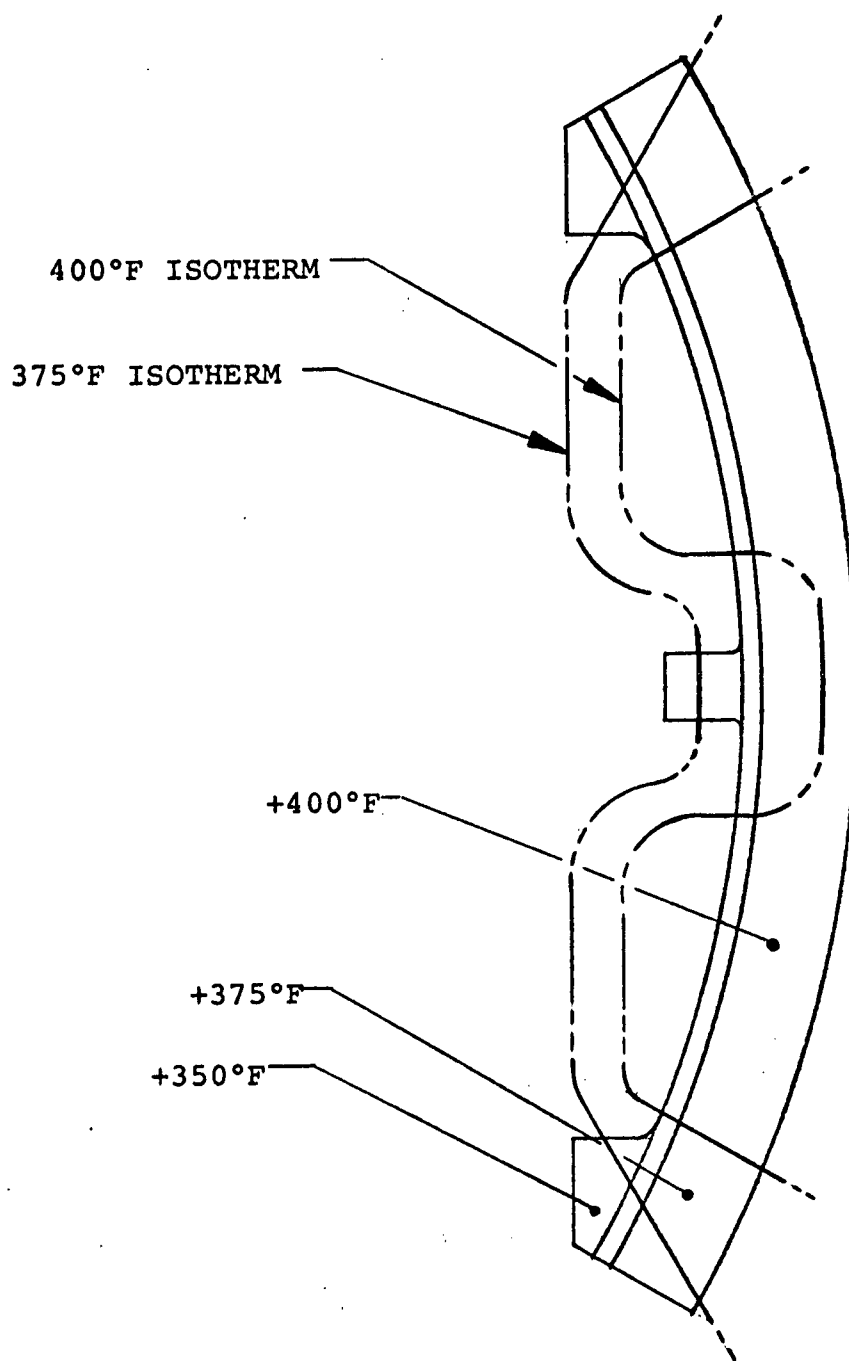
2.4 Phase Three

("--to determine the feasibility of using high temperature resin systems in conjunction with continuous dielectric curing and forming techniques. Use of Polyquinoxylene, Polyimide, Polyphenylquinoxylene, or other appropriate systems -- capable of service at temperatures of at least 600°F").



PHASE ONE TEMPERATURE CONDITIONS

DIAGRAM 1



PHASE TWO TEMPERATURE CONDITIONS

DIAGRAM 2

- 2.4.1 It had been originally intended that we would use either polyquinoxylene or polyphenylquinoxylene for this phase. Both of which were offered, in experimental quantities, by Whittaker's Narmco Research and Development Division. Upon investigation it developed that virtually nothing was yet known about the dielectric characteristics of either of these resins, which would have necessitated an initial research program to determine their respective loss characteristics before proceeding with the actual task of pultruding a length of straight (graphite) hat section. The added cost of that preliminary work, plus the expense of the resins themselves, exceeded - by far - our budget for this phase. Thereby effectively eliminating any serious considerations for using either polyquinoxylene or polyphenylquinoxylene of this program.
- 2.4.2 Whittaker's Narmco Materials Division then submitted new data on their new X5702 high temperature epoxy as a candidate for this Phase III requirement. Preliminary test data showed respectable strength retention to $>500^{\circ}\text{F}$, using "Modmore" Type II graphitic. Flexural strengths reduced from 200,000 psi @ 350°F to only 181,000 @ 500°F , after 500 hours aging at those temperatures. Similarly, flexural modulus showed $19.6 \text{ psi} \times 10^6$ @ 350° and $18.1 \text{ psi} \times 10^6$ @ 500°F . Horizontal shear, which is a most important property in these particular applications, came in at 8,380 psi @ 350°F , but which increased to 9,270 psi @ 500°F . Also after 500 hours aging, at temperature.
- 2.4.2.1 After obtaining NASA's concurrence that this resin showed sufficient promise and capability to be used for the contract's Phase III requirement, an order was placed on September 15, 1971 with Whittaker for 5 pounds of Modmor Type II, impregnated with the new X5207 epoxy resin.
- 2.4.2.2 When three months had gone by it developed, as with many newly developed products, that Whittaker was having considerable trouble reproducing their X5207 resin to its original properties. Whittaker's failure to deliver on schedule was, of course, delaying completion of Phase III. It was decided, therefore, to investigate other suitable resins, while giving Whittaker another 30-day extension of time.
- 2.4.2.3 We first looked at General Electric's "Gemon L" polyimide. G.E.'s local representative - a Mr. K. A. Tennison - offered to see if their normal 6% volatiles could be reduced to something in the order of $<1.5\%$, in order to reduce the known tendency for vapor-phase "blowing" in both dielectric preheat and through-die molding during the pultrusion process. Upon checking back, it turned out that General Electric had summarily dropped their highly-touted "Gemon" polyimide line. And no one at G.E.

knew anything about our request for a resin with lower volatile content. Therefore, since this did not appear to be a situation likely to produce tangible results, we abandoned that particular resin.

- 2.4.2.4 We next sought to obtain a trial quantity of Type II graphite, impregnated with TRW's "P 13 N" polyimide. Upon inquiry to TRW's Robert Vaughn it developed they had just turned P 13 N over to Ciba-Geigy for distribution. The next follow-up step divulged that Ciba-Geigy's Jack Powers and Mike Lutkus were not yet exactly up to speed, technically, on their new resin acquisition and couldn't offer much help, at that particular time at least.
- 2.4.2.5 Further casting around for suitable polyimides brought us to Monsanto's "Skybond 703". Which, after lengthy consultations with Monsanto's Dr. Irving Serlin, seemed to qualify on all counts, performance-wise. And, in addition, appeared to be considerably more forgiving, process-wise. Which would be a definite asset in our attempts to convert, directly, resins designed for bag- or autoclave-molding (excess resin & volatiles) to direct pultrusion. Arrangements were made with Whittaker's Narmco R & D Division to impregnate their Modmor II graphite tow with Skybond 703 and drum wind it - to provide 5.60 in. wide tape, which is the approximate developed surface width of the specified hat section.
- 2.4.3 Whittaker's long-existing order for X5207 epoxy-impregnated graphite 3-inch tape - 7 tows to the inch for better packing density - was cancelled on January 13 for failure to deliver. This was replaced - nearly three months later - by the drum-wound Skybond 703, 5.60-inch wide graphite tape from Narmco R & D (Ref. 4.10).
- 2.4.4 The first pultrusion run with this polyimide resin didn't come off well at all. We had thought that polyimide's known capacity for generating excess volatiles could be circumvented by passing the assembled tape through a circulating hot air tunnel, prior to dielectric preheating, and prior to final die cure. Even with all that pre-heating, the run had to be aborted due to rather violent back-boiling of the resin at the final curing-die's entrance. The next attempt sought to remove initial volatiles (>16%) by coiling the pre-assembled tape and placing it in an air-circulated oven for ~4 hours at 210°F. Upon uncoiling (which had to be done very carefully, of course, because the layup had then become quite stiff and boardy) the assembled tape was fed, almost immediately to avoid moisture pickup, through a standard Glastruder - borrowed from inventory for the purpose - but at a very slow throughput rate. This run proved to be quite successful on all counts.

- 2.4.5 Since we had (obviously) ordered more stock (x 3.5) than would be required to make only the required single 5-foot length of straight graphite/polyimide hat section, there was sufficient stock left for one more run. This one - sans the Teflon slip tapes used on the previous run which, again, wrinkled - went very well. Both lengths were placed on the existing metal postcure fixture and transported to Hitco for postcure. That postcure schedule consisted of beginning at 400°F and increasing 50° after each four hours, ending at 600°F; a total of twenty-hours. (Ref. 4.10). Shipment of (both) the deliverable graphite/polyimide lengths of hat section (only one was required) concluded Phase III of the contract.

2.5 Phase Four

This phase was added to the original contract for the purpose of obtaining an additional quantity (four) of 12-foot lengths of straight graphite/epoxy hat section - for further test work and added experience in pultruding - and one length of curved section -- but this time from graphite epoxy. Which had not been attempted before. Phase IV was, therefore, divided into two sections:

("Utilizing tooling developed under Phases I and II, fabricate the following pultrusion components, using Modmore Type II graphite fiber, 7 tows per inch, impregnated with Whittaker Corporation 5206 hot-melt epoxy:

- (1) 4 - 12' lengths of graphite-epoxy hat stiffener (48' min. total)
- (2) 1 - 12' arc length of curved (10'R) hat stiffener")

- 2.5.1 Laying up the interspersed 0° and ± 45° graphite/#5206 epoxy tapes into 14-foot lengths and thereafter pultruding them to produce 12-foot (net) straight lengths of hat section had, by this time, become nearly a routine operation. Upon receipt of the additional order or Modmor II graphite/5206 epoxy (3-inch-wide) tape the required four lengths were run out. These successive runs were made on an every-other-day basis, with a day and a half devoted to hand laying -up the tape assembly and a half day assigned to adding on leaders and setting up the Glastruder to make the run. Each individual run, however, consumed less than a half hour, startup to runout. Reference to Fig.10 on the following page will show the four lengths of hat section just prior to shipment. This completed Item 1 of Phase IV.

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FIGURE 10 FOUR 12-FOOT LENGTHS OF GRAPHIE/EPOXY HAT SECTION

- 2.5.2 Item 2, of this phase, called for (now) pultruding a 10-foot-radius curved hat section - from graphite/epoxy. All our experience, to that point, had - as has been discussed - been with wet-impregnate woven glass fabric, prepreg rovings/epoxy tapes (but sandwiched between prepreg glass fabrics), etc. As the result of that experience, it was felt that all-linear-tow graphite tapes, when assembled into a multi-layered strap, would probably behave rather typically. Such proved to be not the case.
- 2.5.3 It was anticipated that a potential problem would exist in curving a hat section - inwardly, so to speak, where the hat section's channel is on the inner radius of a circle - resulting from that portion's shorter developed length than its flanges. Steps were taken during those initial runs, using fabric, to smooth the section's cap just prior to its entering the die, in a manner to prevent excess material from bunching and accumulating into a wave. Whereas this technique worked reasonably well with woven fabrics, it failed to repeat that action on all the linear elements in unidirectional-tow graphite tape. There being no cross-ties, as in woven fabric, each individual tow - as inner-radius excess length accumulated - heaved up out of its laminar position and (along with its $\pm 45^\circ$ tape layers) caused splits, fold-overs and twists. (Ref. 4.13)
- 2.5.4 The first such run, of curved hat section, ended after only a few inches had been produced because the assembled tapes began bunching at the die's entrance and jammed. It was deduced, somewhat optimistically as it turned out, that the dielectrically-preheated graphite/epoxy had been sufficiently softened and relaxed as to cause it to fluff; lose its slight compaction achieved during layup. This would, from experience, result in the die's lead-in ("fishmouth") taper being too abrupt; not allowing sufficient time for the built-up layers to be "ironed" into their proper compacted state. To test this theory, the die was simply shimmed apart, between lands, at its entrance end. Which resulted in producing a two-step taper; the initial abrupt lead-in plus a finer, die-length narrowing down of the die's parallel (horizontal only) faces.
- 2.5.4.1 This did allow, on the next attempt, a curved length of about five feet to be produced before the in-feeding material again jammed. But that presumed "fix" turned out to be erroneous because, on checking the thus-produced hat section's general dimensions, it was observed that both cap and flanges had thickened, from the specified .187 in. to roughly .230 in. In reconstructing what had happened, it became apparent that by resorting to an extended length of progressive taper, the wedging force had then become

very high, which actually bent the die's steel support yoke. Remachining a heavier yoke to replace the original only succeeded in transferring the problem back to its original mode; the in-feeding material bunched and jammed at the die entrance after only a foot or so of stock had been pultruded. It was observed that the dielectrically preheated combined tapes, being thoroughly relaxed and quite limp, tended to "fall" out of place as they neared the curing die. This was even further aggravated by the difference between pathlengths - flanges to cap - which had been progressively absorbed, so to speak, when pultruding fabrics and fabrics/roving combinations during the previous Phase II runs.

- 2.5.5 From this performance it can be reasonably predicted that the addition of interspersed layers of woven graphite fabric to the layup would tend to restrain the buckling of individual graphite tows, by exerting a membrane effect on their alternate layers, thereby inhibiting their vertical displacement. However, because this particular hat-section's configuration results in caps and flanges being twice as thick as its side webs - .187 vs. .094 in. - any one-to-one interleaving of woven fabric with linear-tow tape would result in the side webs being constituted solely from fabric, which is somewhat less desirable from a stress standpoint. The other alternative would be to employ fabric, woven from graphite, which is oriented $\pm 45^\circ$. This would be an ideal arrangement for the (shear) webs, of course, and nearly as efficient in cap and flanges, where the fabric would be interspersed with longitudinal-tow layers.
- 2.5.6 Since it seemed reasonably possible that, by encasing those loose graphite tows in surface tapes of woven graphite fabric they could be held together long enough to get the combined laminate into the die, a re-order (the fourth) of graphite/epoxy tape was made. There were also several theories regarding different techniques which might ease this problem.
- 2.5.7 That last attempt resulted in complete failure. The two surface layers of woven graphite fabric did nothing to contain the bulky, layered layup.

- 2.5.8 The problem can be described as being due to the softening and general relaxation of all individual tow elements as the in-feeding layup nears the heated die. As radiated heat begins to warm that area of the laminate just upstream from the die, the epoxy resin softens sufficiently to cause individual tows to sag out of position. This is further aggravated because the hat section is positioned "on edge" as it enters the die, allowing a number of the 0° tows to fall loose and hang in catenaries, which are sometimes several inches in length. It was for this reason we added surface layers of woven graphite fabric, hoping to support the tows and prevent them from falling loose.
- 2.5.8.1 A further fix was attempted which consisted of hand stitching the laminate together with graphite thread, hoping to hold all the individual tow elements together - between fabric surfaces - as the laminate entered the die. But the tows merely dislodged themselves between longitudinal stitched seams. And cross-stitching couldn't be employed due to the need for some shifting - in any laminate over a curved surface - to compensate for the longer geodesic length of the hat section's flanges relative to its cap.
- 2.5.9 Since the problem is really one of gravity, wherein the individual tows literally "fall" out of place when the resin softens, this could be avoided by turning the pultrusion equipment on its side, so to speak, wherein the laminate would enter over the top of vertically-oriented arcuate puller sectors. Instead of their present horizontal position. This would increase, rather substantially, the cost of the equipment. And would obviously necessitate a >25 foot ceiling height.
- 2.5.10 It is still believed possible to pultrude a graphite/epoxy circular hat section on the present equipment through the use of a 1:1 interleaving of fabric between each lamella of graphite-tow tape. This should best be done with graphite fabric, of course, but costs for this material are still extremely high. If a glass or PRD-49 fabric is used, net strengths will be reduced as the rule of mixtures would indicate.
- 2.5.11 We are indeed pleased to have been able to produce the four 12-foot lengths of (straight) graphite/epoxy hat sections. And with no problems. But our failure to produce a circular hat section from the same material disturbs us very much. Five separate runs were made in an effort to produce a successful part, which, due to

the high cost of graphite/epoxy tape, caused us to overrun our (fixed price) budget by 289%. Which is reason enough to admit that we obviously have not yet solved the problem of pultruding circular structural profiles from all-epoxy/graphite-tow materials. (Ref. 4.16).

3.0 CONCLUSIONS AND RECOMMENDATIONS

3.1 Conclusions

- 3.1.1 From the work undertaken on this project, to develop continuous forming and curing techniques for the continuous production of straight and circular structural shapes from composite materials, it has been demonstrated that the pultrusion technique - for aerospace substructural shapes - offers substantial and significant economies over conventional methods. Those economies are largely the result of rather drastically reduced labor costs, over hand layup. In addition, corollary advantages are obtained in the forms of structural uniformity and consistency, over the length of a structural section as well as from part to part. Even further, recent investigations have shown that mechanical properties, of pultruded rods for example, are increased by some 7% across the board, but also flexural modulus and tensile ultimates were increased by as much as 11 and 16%, respectively. This is due solely to the collimating effect of continuously drawing linear filaments or tows into a die, which tends to automatically align and rearrange them until they find their own, most comfortable, and therefore consistent, packing arrangement. Something which is most difficult to achieve in hand layup - if at all.
- 3.1.2 In pultruding - straight - structural shapes and profiles from advanced composites, the necessary compromises of, and considerations for, the process itself are comparatively few. Laminated sections which incorporate lamellas of linear-filament tapes of biased orientation - $\pm 45^\circ$'s, for example - must always have those angle-ply layers covered by a surface lamella of axially (0°) oriented material. This, to prevent off-axis filaments from being skewed out of position by friction against die surfaces as the laminate is being pulled through. The only other compromise of any magnitude is the need for fairly generous corner radii to prevent resin-starving (sharp inside corners) or resin-rich edges (outside corners). The latter, however, is a rather classic design precaution for all pultrusions and is not a consideration applicable only to advanced composites.
- 3.1.3 Epoxy matrices can be pultruded with very nearly the same process parameters and conditions as the (industrially classic) polyesters, except - because they are far better adhesives, per se - they tend to try to adhere to the metal faces of pultrusion dies, creating a light scum on die surfaces. This reduces product surface finishes from

one of typical shiny appearance to one of matte; sometimes even quite dull. However, except where an individual flake of resin has adhered to the die face - causing a plowed groove in the emerging stock - or when uniformly aligned tows, in particular, leave light resin ridges on the length of a die face, resulting in a parallel series of shallow scores on the emerging stock, the customary dull surface attributable to epoxy resins is probably not particularly detrimental. Or at least it hasn't been shown to be in preliminary tests. Conversely, a dull finish would be expected to offer a better surface for adhesive bonding.

- 3.1.3.1 It should be noted, here, that commercial pultrusions are sometimes required to have a superficial resinous coating applied, either to enhance their appearance (an enamel-like colored finish) or to give them an even higher resistance to the corrosive effects of specific environments. Fortuitously, since pultruded stock emerges quite hot - $\sim 340^{\circ}\text{F}$ - the immediate application of a coating resin will be cured, on line, by that residual heat. Such coatings are applied in several ways; passing the pultrusion through a (cooled) pot of liquid resin, using silicone rubber bushings at entrance and exit points to retain the resin; and, similarly, passing the hot stock through a special fluidized bed device, where dry colloidal powders of resin are swirled against the surface until they have accumulated a deposited layer of 2-4 mils. The latter is becoming more favored as new and varied powered resins become available.
- 3.1.4 Concerning the continuous production of straight pultrusions, for aerospace applications and employing advanced composite materials, it can be concluded that there are no real problems. Being a straightforward translation of commercial pultrusion techniques, that quite large body of existing experience (>23 years) serves as a reservoir of practical knowhow from which to handle the production and operating requirements, leaving only those considerations for adapting somewhat different materials to this existing industrial process. Which, to this time, has been fairly easy.
- 3.1.5 The making of circular, pultruded shapes, originally thought to be somewhat chancey, was proven - by this contract - to be entirely feasible. Circularly formed hat-section shapes were produced by this method, on specially adapted, ancillary pultrusion equipment. The

pultrusion principles still remain, but with rotating tools serving as both the essential "pullers" and one, opposite, forming-die face. The use of two, 45° ring sectors allows the continuous production of any practicable number of rings, accumulating in the form of an upwardly growing spiral. Which can later be cut into individual rings. With only the one splice joint.

- 3.1.6 Although the upstream end of this (new) circular pultrusion machine resembles any standard "Glastruder" production machine - materials feed-in creels, tape spools, shape-preforming plates, resin impregnation, dielectric heating cavity and rf generator, etc. - from that point forward the conventional pultrusion aspects became entirely different. This is perhaps a contributing reason for the number of difficulties encountered; we could not call upon our existing experience to help us with pultruding circular shapes. Because there isn't any.
- 3.1.7 Nonetheless, there are several conclusions to be drawn from this work.
 - 3.1.7.1 It is now generally agreed that the mechanical principles used in the design of the circular forming/pulling arrangements are probably basically correct. But a next-generation machine will need to be modified and upgraded in certain of its areas. More attention must be paid to the heated-rim portion of the arcuate sectors, in order to achieve a more precise control of rim-die temperatures. And - more importantly - seek to flatten the isothermic profiles's present cure-temperature amplitudes. The greatest detractors of heat are at those support points where the structure's radial spokes are connected to each rim. This was anticipated, but insufficient heat-blocking was provided. And, additional - thicker - insulation should be used over all heated surfaces, except the actual rim/die face itself. This, to both level the die's mean temperature and reduce, substantially, the net wattage being consumed.
 - 3.1.7.2 It has been established that woven glass fabric tapes, either preimpregnated or on-line wet impregnated with epoxy, can be pultruded to produce a formed ring. As well as polyester-impregnated fabrics and glass rovings, as has been reported. It has been further demonstrated that dry woven-fabric tapes - themselves wet impregnated, in line - can be drawn in, simultaneously, with preimpregnated roving tapes to produce an epoxy-impregnated ring structure.

- 3.1.7.3 First attempts to pultrude similar hat-section rings from all-graphite-tow laid up tapes resulted in jamming at the entrance to the final curing die. To the extent the bolster yokes restraining the fixed, male die were permanently deformed. Heavier yokes did not relieve that problem. It was subsequently observed that the (already dielectrically preheated) assembled tapes began to become disassociated, internally, as the laminated strap neared the vicinity of the final curing die. This appeared to result from heat being radiated into the strap by the tangentially-approaching, heated die rim. Thereby causing the already preheated material to relax even further, resulting in the majority of individual tows becoming unstuck from one another, and literally, falling apart. Once loose, the tows - presumably due to different levels of tension used during tape-making - would assume varying attitudes, with a large percentage buckling up from their various planar positions and, in some cases, then folding over or sagging down on top of their neighbors. Thereby causing thickened sections, which subsequently jammed the die.
- 3.1.7.4 Various fixes were attempted to shield the approaching laminate from being subjected to radiating heat from the curved die rim, without noticeable success. The level of dielectric preheat was reduced - even turned off - to see if cold tapes would be less susceptible to having their tows become displaced, but this had little effect; those tows on the inside - nearest the hot die rim - continued to buckle and fall out of position.
- 3.1.7.5 These fruitless experiments had consumed nearly 30 pounds of graphite/epoxy tape, already, but it was decided that, notwithstanding this sizeable cost overrun, we would make at least a couple more experimental runs -- using woven graphite fabric on both exterior surfaces to - hopefully - restrain the linear-tow tapes in position. At least long enough to allow the laminate to get into the curing die without fluffing itself apart. Just adding those graphite-fabric surfaces did nothing to ease the situation; the immediately underlying tows still buckled and pushed the fabric away. Attempts to through-stitch the fabric also proved ineffective.
- 3.1.8 From this it can be concluded that considerable further work needs to be done to develop a technique - possibly augmented with quite different layup sequences and/or the interleaving of woven fabrics, perhaps on a one-to-one basis with all-tow tapes - to produce circular shapes from preimpregnated graphite-tow/epoxy tapes.

3.2 Recommendations

- 3.2.1 The pultrusion technique, having established - under this contract - its effectiveness and economy in producing standard structural shapes from both glass and graphite reinforcements, still needs an auxilliary means for laying in, mechanically, off 0°-axis layers. Continuously. The addition of such tape-laying devices - actually, simple tape-laying heads, roughly similar to those we have supplied in the past - would bring the processing cost, of such pultruded structural shapes, down to a labor-cost level comparable to that of industrial pultrusions. Which is currently being figured at only ~1-2¢ a running foot for an operator and his helper. It is our recommendation that serious consideration be given to the obvious need for such upstream, on-line, angle-ply tape laydown devices. Which will, thereby, allow this process - which is inherently continuous - to draw in already-laminated and properly stacked tape layers, of whatever orientation, to allow the equipment to operate properly; non stop. With emerging stock allowed to run out to any (transportable) length before being cut.
- 3.2.2 It is also recommended that further work be undertaken to solve the remaining problems in producing circular shapes from linear-tow-graphite/epoxy tapes. Or, alternatively, determine the necessary material compromises needed to produce such structural rings from combinations of reinforcing materials.
- 3.2.3 Lastly, it is further recommended that a study be conducted to determine which of the presently identified candidate space vehicle applications for pultruded sub-structural shapes are most in need of this production system; to reduce costs and/or weight. And that prototype tooling be authorized for producing structural samples for comprehensive tests to confirm capability to perform as required. Otherwise, it is entirely possible that technical lethargy may very well influence, by default, a proper selection between all of the presently available materials of construction. In which case this newly-developed faculty will probably be simply overlooked.

4.0 REFERENCES & ACKNOWLEDGEMENTS

- 4.1 First Monthly Progress Report, August 18, 1971
- 4.2 Second Monthly Progress Report, September 2, 1971
- 4.3 Third Monthly Progress Report, September 24, 1971
- 4.4 Fourth Monthly Progress Report, October 26, 1971
- 4.5 Fifth Monthly Progress Report, November 24, 1971
- 4.6 Sixth Monthly Progress Report, January 11, 1972
- 4.7 Seventh Monthly Progress Report, January 26, 1972
- 4.8 Eighth Monthly Progress Report, February 29, 1972
- 4.9 Ninth Monthly Progress Report, March 23, 1972
- 4.10 Tenth Monthly Progress Report, May 4, 1972
- 4.11 Eleventh Monthly Progress Report, July 14, 1972
- 4.12 Twelfth Monthly Progress Report, August 1, 1972
- 4.13 Thirteenth Monthly Progress Report, September 12, 1972
- 4.14 Fourteenth Monthly Progress Report, October 4, 1972
- 4.15 Fifteenth Monthly Progress Report, November 6, 1972
- 4.16 Sixteenth Monthly Progress Report, December 14, 1972

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